Alkalinity

EQUIPMENT

Digital titrator

Sulfuric acid titrator cartridge (**NOTE:** sulfuric acid is corrosive and potentially carcinogenic—do not create aerosols or inhale)

Delivery tube

Powder pillows: Phenolphthalein and Bromcresol Green-Methyl Red

Magnetic stir plate

Stir bar

Gloves, goggles, lab coat

Scissors

METHOD SUMMARY

The Hach method for total alkalinity measurement involves titration of a sample with sulfuric acid in the presence of a color indicator. The endpoint is a color change that depends on the final pH of the titrated sample. The method measures total alkalinity in mg/L $CaCO_3$. In some cases, an intermediate measurement of phenolphthalein alkalinity is made.

Two sulfuric acid titrator cartridges are available: 0.1600 and 1.600 N sulfuric acid. The **1.600 N** cartridge is used for alkalinity measurements between 100 and 400 mg/L $CaCO_3$, which is considered the typical analysis for most samples. If the sample is determined to be out of this range, see the Hach Digital Titrator manual, page 39, for correct sample volumes and cartridge selection.

SAMPLE HANDLING

Samples should be brought to room temperature and measured as soon as possible. Samples may be held at 4°C for at least 24 hours.

Typically, 100 ml of sample is required for measurement. However, because sample alkalinity cannot be predicted, extra sample (at least 25 ml) should be available for repeat analyses.

MEASUREMENT OF ALKALINITY

1. Check for freshness of reagents.
2. Check for date of last Quality Control check (in Calibration Log)—if > 3 months, check again (see Quality Control section).
3. Place correct titrator cartridge into digital titrator. Place correct delivery tube into syringe (tip only) and turn delivery knob until fluid comes out of tube. Wipe tube.
4. Measure appropriate volume (typically 100 ml) of sample into 250-ml plastic tri-pour beaker.
5. Place stir bar in sample; set sample on stir plate.

6. Zero the titrator.
7. Add contents of one Phenolphthalein Powder Pillow.
8. If solution turns pink, lower the digital titrator until tube is approximately 1 cm below surface of sample. *(Typically, the solution will not turn pink, and the analyst will skip to step 11.*)
9. Begin slowly adding sulfuric acid with delivery knob, and titrate to a colorless endpoint.
10. Read the number on the titrator (do not zero titrator after this). When measuring in the range of 100 to 400 mg/L, this number equals the actual mg/L phenolphthalein alkalinity as $CaCO_3$ present in the sample. For other measurement ranges, see manual for digit multiplier.
11. Record the phenolphthalein alkalinity (P alk = _____) on data sheet.
12. Add contents of one Bromcresol Green-Methyl Red Powder Pillow. Begin slowly adding sulfuric acid with delivery knob until color change is noted. Proper color endpoint depends on sample composition:

Sample composition	pH at endpoint	color at endpoint
Alkalinity about 30 mg/L	pH 5.1	light greenish blue-gray
Alkalinity about 150 mg/L	pH 4.8	light violet-gray
Alkalinity about 500 mg/L	pH 4.5	light pink
Silicates or phosphates present	pH 4.5	light pink
Industrial waste/complex sample	pH 3.7	see manual

You can use pH change instead of color change as an endpoint—use pH meter to measure.

13. Read the number on the titrator. When measuring in the range of 100 to 400 mg/L, this number equals the actual mg/L total alkalinity as $CaCO_3$ present in the sample. For other measurement ranges, see manual for digit multiplier. Record the total alkalinity.
14. Zero titrator and rinse stir bar before each subsequent sample.

QUALITY CONTROL

Check for method and titrator accuracy monthly. Measure 100 ml of sample in the 100-400 mg/L alkalinity range into 250-ml plastic tri-pour beaker. Titrate to color change for total alkalinity as described above. Snap the neck off of an Alkalinity Voluette Ampule, 0.500 N. Add 100 μL to the titrated sample, and titrate back to the endpoint. Twenty-five titrator digits should be used to return to the endpoint. Do this standard addition three times, keeping in mind that the color endpoint will change as you increase the alkalinity of the sample. Record additions in Calibration Log book on the Hach Alkalinity and Hardness QA Log. Titration should be within 10% of 25.